# Hydromatix

# **Technology Evaluation**

Workplan

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# A. INTRODUCTION

# 1. PROJECT ORGANIZATION

# TITLE AND APPROVAL SHEET

| Department of Toxic Substances Control   | U.S. Environmental Protection Agency |  |  |
|--|--------------------------------------|--|--|
|  |                                      |  |  |
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| W D: 1 I                                 |                                      |  |  |
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#### **DISTRIBUTION LIST**

# Department of Toxic Substances Control

Mr. Tony Luan, DTSC/OPPTD (Primary Decision Maker)

Mr. Dick Jones, DTSC/OPPTD (Project Manager)

Ms. Ruth Chang, DTSC/HML (QA/QC Reviewer)

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# U.S. Environmental Protection Agency

Ms. Norma M. Lewis, U.S. EPA (Technical Project Manager)

Ms. Lauren Drees, U.S. EPA (QA/QC Officer)

# Hydromatix, Inc.

Mr. Greg White, Vice President of Sales and Marketing

#### PROJECT MEMBER DUTIES AND RESPONSIBILITIES

# **Department of Toxic Substances Control**

Assignment Manager Tony Luan has final DTSC authority and oversight of planning

team's activities.

Project Manager Dick Jones is responsible for overseeing implementation of the

Technology Evaluation Workplan (TEW), coordinating project team meetings, ensuring that necessary resources are provided for

planning team decisions, and preparing project reports.

QA/QA Reviewer Ruth Chang is responsible for ensuring the data collection design

meets QA/QC requirements.

Industrial Hygienist Ken Beutler is responsible for worker health and safety.

Project Reviewers Bruce La Belle and Ed Benelli are responsible for providing

technical input.

Planning Team Members All team members are responsible for participating in plan

preparation activities, project meetings and reviewing project reports. Each member of the project team was selected based on

their knowledge, experience, and/or responsibility.

## US Environmental Protection Agency

Project Manager Norma Lewis is responsible for providing U.S. EPA review,

oversight, and implementation of the TEW, and data evaluation of

the TEW reports.

QA/QC Lauren Drees is responsible for providing U.S. EPA QA/QC

review of the TEW and data analyses.

#### 2. PROJECT BACKGROUND

Hydromatix, Inc. has applied to the joint U.S. EPA and Cal/EPA Environmental Technologies Verification Program (ETV) for federal verification and state certification of the Hydromatix 786E System ion exchange regeneration technology. The 786E system is used to remove cations and anions from rinse wastewaters generated during metal finishing operations such as electroplating, cleaning, and anodizing. The regeneration technology consists of a process logic controller (PLC), sensors, and associated plumbing for regeneration of the resins and for collection of the regenerant waste. Regeneration of ion exchange resins consists of a series of acid and base rinses which result in restored resin functionality, but which also produce a waste solution. This waste requires further processing before it can be reused or disposed. The Technology Evaluation Workplan details the activities to be conducted at the Aero-Electric Connector (AEC) facility in Torrance, California in order to evaluate the Hydromatix 786E System.

## **Problem Definition**

Large volumes of wastewater are produced during the regeneration of ion exchange resins. Typically a hazardous waste, this regenerant wastewater constitutes a substantial fraction of the original volume of rinse wastewater treated. Precipitation and clarification is often used as a final treatment because it is able to process large volumes efficiently. This treatment method generally produces waters which meet POTW or NPDES discharge requirements, but also produces a residual sludge which needs further treatment prior to disposal. The large volume of regenerant wastewater requiring precipitation and clarification treatment often precludes the use of evaporation as a treatment method, which could result in zero discharge from the facility.

#### **Problem Resolution**

Hydromatix developed an ion exchange regeneration process for their Model 786E series systems which uses a PLC system to coordinate acid and base rinse water reuse, thereby reducing the volume of regenerant chemicals used as well as the volume of regenerant wastewater produced. This allows the use of evaporation as a final treatment method, which results in a zero discharge facility. The Hydromatix system features packed exchange columns with conductivity meters, electronic logic, and automatic valves to control the regeneration process. By employing reuse of portions of the regenerant rinses as make-up solutions for the next cycle, and by returning other rinses to the feed tank rather than to waste, the system is able to achieve a substantial reduction in the amount of chemicals used as well as in the amount of wastewater produced.

#### 3. PROJECT DESCRIPTION AND OBJECTIVES

The purpose of the Technology Evaluation Workplan is to provide information necessary for the ETV Program verification and California certification evaluation. The central claim made by Hydromatix is that their technology results in a reduced volume of regenerant waste produced. The specific volume, the ratio of gallons of waste produced per cubic foot of resin regenerated, is

small compared to conventional ion exchange systems. This smaller volume allows more waste management options and assists in achieving zero discharge. Hydromatix claims that a minimum of 1 eq/L of ion exchange capacity can be restored while generating a specific volume of no more than 10.5. Thus, the primary objectives of the Workplan are to determine (1) the specific volume of regenerant waste produced, and (2) the cation and anion exchange capacities restored during regeneration. Secondary objectives include providing information for potential end-users and metal reclaimers, and observing the system during normal operating conditions in order to evaluate worker health and safety. Only the specific volume and associated parameters of the Hydromatix system will be evaluated in this Workplan; no other competing ion exchange technologies will be investigated, and operating cost data will not be collected.

# B. FIELD TESTING AND DATA ACQUISITION

Installed in October 1997, Aero-Electric has one of the oldest 786E systems in operation. AEC reports the system has been regenerated more than 200 times with no detectable degradation in resin performance. The system contains 18 cubic feet each of Purolite PFC-100H cationic and Purolite PFA-300 OH anionic resins. Rinse wastewater flow to the system is typically around 35 gallons per minute.

Four separate test runs using the same cation and anion resin columns will be conducted at AEC. Each test run will consist of a complete treatment cycle including column exhaustion and regeneration. The field data to be collected from each test run is listed in Table 1, Field Monitoring, Sampling, and Analytical Methods.

#### 1. Regenerant Waste Volume Produced

During each test run the volume of regenerant waste produced will be measured with an inline flow totalizer. The regenerant waste volume will be correlated with the capacity restored during that regeneration. The range, average, and confidence interval for the regenerant waste volume produced will be determined from the four runs.

## 2. Cation and Anion Exchange Capacities Restored

The cation and anion exchange capacities restored during regeneration will be determined by sampling the regenerated resin directly. Samples of the resins will be collected using standard industry methodology, which typically involves inserting a PVC pipe or other device into the resin beds from above; when withdrawn the tube retains a core sample of the resin material. To ensure a representative sample through the bed, the pipe will be inserted through the full six foot bed depth. The capacities restored, or percent regenerated, will be determined analytically at the resin manufacturer's laboratory (PuroliteUSA, Bala Cynwyd, PA).

The capacities restored to the columns will also be checked by performing a mass-balance on incoming rinse wastewater, product DI water, and regenerant waste. Using volume measurements, the concentrations of ions found in these streams will be converted to equivalents, and totaled, providing the number of equivalents entering and leaving the system. A charge

Table 1 - Field Monitoring, Sampling, and Analytical Methods

| Туре                             | Parameter(s)  | Frequency     | Location  | Method(s)  | Containers<br>(Storage Limits)   |
|----------------------------------|---|---------------|---|--|--|
| Rinse<br>Wastewater              | Volume  | Each test run | Feed line from collection tank  | Inline flow totalizer                                  | Recorded on site   |
|                                  | Flow rate   | Daily         | Feed line from collection tank  | Inline flow totalizer                                  | Recorded on site   |
|                                  | Al, B, Cu, K, Na, Ni, Zn  | Each test run | ISCO Automatic<br>Sampler at collection<br>tank                               | U.S. EPA Methods<br>3010A, 6010B                       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months)                                   |
|                                  | NH <sub>4</sub> <sup>+</sup>  | Each test run | ISCO Automatic<br>Sampler at collection<br>tank                               | U.S. EPA Method<br>350.2                               | 1000 ml P or G,<br>H <sub>2</sub> SO <sub>4</sub> , pH<2<br>(350.2 28 days)                      |
|                                  | Cl <sup>-</sup> , F <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , NO <sub>3</sub> <sup>-</sup> , PO <sub>4</sub> <sup>3-</sup><br>Total dissolved solids,<br>Specific conductance,<br>Alkalinity | Each test run | ISCO Automatic<br>Sampler at collection<br>tank                               | U.S. EPA Methods<br>300.0, 9050A, 310.1,<br>160.3      | 1000 ml P or G,<br>4 C, no preservative<br>(300.0 48 hours)<br>(310.1 7 days)<br>(9050A 28 days) |
|                                  | pH  | Each test run | ISCO Automatic<br>Sampler at collection<br>tank                               | U.S. EPA Method<br>150.1                               | Measured on site at time of collection   |
| Rinse<br>Wastewater<br>Duplicate | Al, B, Cu, K, Na, Ni, Zn  | Each test run | ISCO Automatic<br>Sampler at collection<br>tank                               | U.S. EPA Methods<br>3010A, 6010B                       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months)                                   |
| Product DI<br>water              | Al, B, Cu, K, Na, Ni, Zn  | Each test run | Grab sample from<br>Product DI water<br>pipe sample port                      | U.S. EPA Methods<br>3010A, 6010B                       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months)                                   |
|                                  | NH <sub>4</sub> <sup>+</sup>  | Each test run | Grab sample from<br>Product DI water<br>pipe sample port                      | U.S. EPA Method<br>350.2                               | 1000 ml P or G,<br>H <sub>2</sub> SO <sub>4</sub> , pH<2<br>(350.2 28 days)                      |
|                                  | Cl <sup>-</sup> , F <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , NO <sub>3</sub> <sup>-</sup> , PO <sub>4</sub> <sup>3-</sup><br>Total dissolved solids,<br>Specific conductance,<br>Alkalinity | Each test run | Grab sample from<br>Product DI water<br>pipe sample port                      | U.S. EPA Methods<br>300.0, 9050A, 310.1,<br>160.3      | 1000 ml P or G,<br>no preservative<br>(300.0 48 hours)<br>(310.1 7 days)<br>(9050A 28 days)      |
|                                  | pН  | Each test run | Grab sample from<br>Product DI water<br>pipe sample port                      | U.S. EPA Methods<br>150.1                              | Measured on site at time of collection   |
|                                  | EC reading  | Daily         | Sensor is in the effluent pipe; display is on the panel                       | Rosemont Analytical<br>Solu Comp Model<br>SCL-C-002-M2 | Recorded on site   |
| Cationic<br>Regenerant<br>Waste  | Volume  | Each test run | Line from cationic column to neutralization tank                              | Inline flow totalizer                                  | Recorded on site   |
|                                  | Al, B, Cu, K, Na, Ni, Zn  | Each test run | ISCO Automatic<br>Sampler installed at<br>Regenerant waste<br>collection line | U.S. EPA Methods<br>3010A, 6010B                       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months)                                   |
|                                  | NH <sub>4</sub> <sup>+</sup>  | Each test run | ISCO Automatic<br>Sampler installed at<br>Regenerant waste<br>collection line | U.S. EPA Method<br>350.2                               | 1000 ml P or G,<br>H <sub>2</sub> SO <sub>4</sub> , pH<2<br>(350.2 28 days)                      |

Table 1 - Field Monitoring, Sampling, and Analytical Methods, continued

| Туре                               | Parameter(s)  | Frequency     | Location  | Method(s)                      | Containers<br>(Storage Limits)                                 |
|------------------------------------|---|---------------|---|--------------------------------|--|
| Anionic<br>Regenerant<br>Waste     | Volume  | Each test run | Line from anionic column to neutralization tank                               | Inline flow totalizer          | Recorded on site   |
|                                    | Cl <sup>-</sup> , F <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , NO <sub>3</sub> <sup>-</sup> , PO <sub>4</sub> <sup>3-</sup> | Each test run | ISCO Automatic<br>Sampler installed at<br>Regenerant waste<br>collection line | U.S. EPA Method<br>300.0       | 1000 ml P or G,<br>no preservative<br>(300.0 48 hours)         |
| QA Travel<br>Blank- Metals         | Al, Cu, Ni, Zn  | Each test run | Prepared at So Cal<br>HML   | U.S. EPA Method<br>6010B       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months) |
| QA Travel<br>Blank-<br>Anions      | Cl <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , PO <sub>4</sub> <sup>3-</sup>   | Each test run | Prepared at So Cal<br>HML   | U.S. EPA Method<br>300.0       | 1000 ml P or G,<br>(300.0 48 hours)                            |
| QA Spike-<br>Metals                | Al, Cu, Ni, Zn  | Each test run | Prepared at So Cal<br>HML   | U.S. EPA Method<br>6010B       | 1000 ml P or G,<br>HNO <sub>3</sub> , pH<2<br>(6010B 6 months) |
| QA Spike-<br>Anions                | Cl <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , PO <sub>4</sub> <sup>3-</sup>   | Each test run | Prepared at So Cal<br>HML   | U.S. EPA Method 300.0          | 1000 ml P or G,<br>(300.0 48 hours)                            |
| Cationic column resin              | Operating capacity remaining, regeneration efficiency   | Each test run | Cationic column   | Purolite Laboratory methods    | 1000ml P or G,<br>sample must be kept moist                    |
| QA cation<br>resin Travel<br>Blank | Operating capacity, regeneration efficiency   | One sample    | Fresh Cationic resin from Purolite  | Purolite Laboratory<br>methods | 1000 ml P or G, sample must be kept moist                      |
| Anionic column resin               | Operating capacity remaining, regeneration efficiency   | Each test run | Anionic column  | Purolite Laboratory methods    | 1000 ml P or G, sample must be kept moist                      |
| QA anion<br>resin Travel<br>Blank  | Operating capacity remaining, regeneration efficiency   | One sample    | Fresh Anionic resin from Purolite   | Purolite Laboratory<br>methods | 1000 ml P or G, sample must be kept moist                      |
| Acidic regenerant                  | Volume  | Each test run | Line from acid tank to cation column  | Inline flow meter              | Recorded on site   |
|                                    | HCl   | Each test run | Grab sample from<br>Acid make-up tank   | U.S. EPA Method<br>305.1       | 1000 ml P or G<br>(305.1, as short as practically<br>possible) |
| QA Acid<br>standard                | нсі   | One sample    | Prepared at So Cal<br>HML   | U.S. EPA Method<br>305.1       | 1000 ml P or G<br>(305.1, as short as practically<br>possible) |
| Basic<br>Regenerant                | Volume  | Each test run | Line from acid tank to cation column  | Inline flow meter              | Recorded on site   |
|                                    | NaOH  | Each test run | Grab sample from<br>Base make-up tank   | U.S. EPA Method<br>310.1       | 1000 ml P or G<br>(310.1, as short as practically<br>possible) |
| QA Base<br>standard                | NaOH  | One sample    | Prepared at So Cal<br>HML   | U.S. EPA Method<br>310.1       | 1000 ml P or G<br>(310.1, as short as practically<br>possible) |

Some samples may exhibit matrix interference defects. The lab will be notified of high ionic concentration samples. Sample preparation could include such steps as removing interfering ions, additional sample dilutions, or analysis using the method of standard additions.

balance will be performed to indicate if ions were undetected or the analysis is otherwise unreliable<sup>1</sup>.

#### 3. Rinse Wastewater Volume Treated

The rinse wastewater volume treated will be measured with an inline flow totalizer.

#### 4. Masses of Acid and Base Volume Consumed

The masses of acid and base used per regeneration will be determined by monitoring the volumes of acid and base solutions applied to the columns during regeneration, combined with measurements of those solutions for concentration. The flows from the acid and base tanks will be measured with an inline flow totalizer.

## 5. Masses of Metal Species in the Regenerant Waste

The concentrations of cations in the regenerant waste will be determined for mass balance calculations and to provide information for potential end-users and metal reclaimers. The results will be used with the volume measurements to calculate the masses in the regenerant. The range, average, and confidence interval for the masses of metal species in the regenerant waste will be determined from the four runs.

# 6. Product DI Water Quality

The electrical conductivity (EC) of the product DI water is currently monitored by an existing conductivity sensor/meter (Rosemont Analytical Solucomp model) in the effluent pipe. Hydromatix reports that at the beginning of each treatment mode, the EC is about 0.3 microsiemens/cm ( $\mu$ S/cm), and that the EC set point for the end of the treatment mode is normally chosen to be about 20  $\mu$ S/cm. When the sensor/meter indicates the EC is greater than or equal to the set point, the treated water EC indicator light on the panel illuminates and the rinse wastewater flow is re-directed to a fresh set of columns. EC readings will also be recorded daily from the panel display.

As a check on the sensor/meter indication, and to determine which ions contribute to the EC, the product DI water will be analyzed for the same constituents as the rinse wastewater. The range of empirical multipliers for conversion of  $\mu$ S/cm to meq/L is reported<sup>2</sup> to be about the same for divalent and monovalent ions (the influent is primarily divalent and monovalent ions and

<sup>&</sup>lt;sup>1</sup> An acceptable range for the difference between anion and cation sums has been empirically established as  $\Sigma$  anions -  $\Sigma$  cations =  $\pm$  (0.1065 + 0.0155  $\Sigma$  anions)

Franson, Mary Ann, <u>Standard Methods for the Examination of Water and Wastewater</u>, 16<sup>th</sup> ed., Am. Pub.Health Assoc., Washington, DC, 1985, p. 32. Values falling outside the limits set by this equation indicate that at least one of the determinations should be rechecked.

<sup>&</sup>lt;sup>2</sup> Tchobanoglous and Schroeder, pg. 92

effluent is mostly monovalent ions). Thus, the results from the EC and constituent analyses of the treated rinse water can be compared. Four samples will be collected and measured for electrical conductivity, one at the end of each treatment run, when the set point has been reached. The range, average, and confidence interval for the EC and the ionic constituents in the product DI water will be determined from the four runs.

# 7. Worker Health & Safety

Questionnaire responses (Appendix B), telephone interviews, onsite observations, and review of the generic Hydromatix 786E system drawings will be used to assess the risks posed to worker health and safety. These inquiries and observations will indicate whether accidental releases due to the failure of piping, valves, or pumps, are possible, and whether routine contact with the system results in worker exposure. The risk of exposure can be reduced by operators following safe operating procedures including adherence to the

VAPOR PHASE REGENERANT DISCHARGE REUSE ANIONIC REGENERANT CATIONIC REGENERANT NEUTRALIZATION TANK TANK TANK **EVAPORATOR** 6% HCI 6% NaOH UNITS FLOW METER RESIDUAL SLUDGE FLOW METER ISCO SAMPLER FLOW METER 8 DOWNFLOW REGENERATION C1 CATION A1 ANION C2 CATION A2 ANION GAC UPFLOW SERVICE FLOW CONDUCTIVITY PROBE METER ISCO SAMPLER UV DISINFECTION FILTER PRODUCT DI WATER WASTE RINSEWATER STORAGE TANK FEED TANK PROCESS LINE RINSE TANKS FEED PUMP 35 gpm RINSE WASTEWATER  ${\mathfrak Q}$ 

Figure 1 Hydromatix System Diagram

Installation, Operation & Maintenance Manual for the system at Aero-Electric Connector, and by wearing appropriate personnel protective equipment during operation and servicing of the system. If any releases occur during the field testing the operators' responses to them will be recorded.

### 8. Field Instruments, Equipment and Test Method

#### Flow Sensor

Signet model 515 Rotor-X Flow Sensors will be installed at the following locations: the rinse water feed line between filter and carbon filter, the line between acid tank and cation column, the line between caustic tank and anion column, and the regenerant waste line between the columns and the neutralization tank. Manufacturer's recommendations for equipment installation shall be followed in all cases in order to achieve the maximum accuracy for the instruments. The recommendations include provisions for pipe run lengths before and after the installation locations, and equipment orientation and placement upon the piping. The individual calibration certificate for each flow sensor shall be retained as original field data. The sensors will be removed from the system after the testing is complete and returned to the manufacturer for recalibration testing. The results of the second calibration test at the manufacturer shall be retained as original field data.

#### Flow Totalizers

Signet flow totalizers model 8550, 9010, or equivalent, will be installed to record readings from each flow sensor.

#### **Automatic Sampler**

An ISCO Automatic Sampler Model 6800 will be installed and set to sample periodically at the following locations: at the rinse wastewater feed tank, and the regenerant waste line between the columns and the neutralization tank. The volume collected during each collection period will be determined by dividing the desired total sample size by the number of days, then by work hours per day, and then by the number of collection events desired per hour. The feed tank will be sampled directly through the manhole port in the top of the tank. The regenerant waste line will require installation of a Pressure Reduction Valve (ISCO Model SPA 1081), a Three-way Valve (ISCO Model SPA 1082), and a Relay Contact (ISCO Model SPA 665).

The most desirable sampling scheme is to collect the maximum volume, two-and-a-half gallons, over the course of the several day-long treatment period. In this situation the collection period may be 10, 12, or 18 hours per day, for as many as three, four, or five days, depending upon the production workload and consequent ionic loading supplied to the resin columns. Thus, the sample volumes and frequency must ensure that the total volume collected does not exceed the maximum; reaching this volume will trigger a float switch and stop the collection, thereby failing to collect a representative sample from the entire production run. Consequently, a sufficient sampling protocol may be to assume 18 hours of production per day, for five days.

Sampling every 15 minutes would then dictate individual sample volumes of approximately 26 mLs, which is an appropriate volume for this type of sampling device. To prevent sampling during hours when there is no production the sampling unit will be triggered externally using a flow signal generated by the flow transmitter. Once programmed, the ISCO sampler will be calibrated with a graduated cylinder by DTSC personnel.

## **Resin Sampling Probe**

PVC pipe of suitable length and diameter (approximately seven feet long, one-half inch in diameter), or equivalent soil sampling device.

## **Electrical Conductivity Meter**

The existing Rosemont Analytical, model SCL-C-002-M2 electrical conductivity sensor/meter, readable to the nearest 0.1  $\mu$ S/cm will be used to measure the electrical conductivity of the treated rinse water. Rosemont Analytical reports the accuracy and precision at 0.1  $\mu$ S/cm. The sensor/meter readings will be checked using laboratory analysis and the results will not be used for statistical calculations.

### pH Meter

A pH meter will be used to measure samples directly onsite at the time of sample collection. The unit will be of sufficient quality to adhere to the requirements of U.S. EPA Method 150.1, including a provision for two-point calibrations. The unit will be calibrated each day that readings are collected.

#### **Test Method**

Four consecutive test runs will be performed, with each test run defined as beginning with a treatment mode. Use of a single cation and anion exchange bed pair are specified for each test run. DTSC representative(s) will be present to oversee the test runs and perform sample collection.

The testing procedures during each of the test runs will involve the following steps:

- 1. Note the flow totalizer reading at the start of the rinse waste treatment mode. Start treatment mode and begin collecting samples from the rinse wastewater feed tank using an ISCO automatic sampler.
- 2. Note EC readings daily during the treatment run.
- 3. Upon reaching maximum EC set point, "pause" system, note flow totalizer reading, stop ISCO sampler, retrieve composite sample, and collect grab sample from product DI water line. Take pH reading.

- 4. At the conclusion of the treatment mode, while the system is set to "pause", note the acid solution flow totalizer reading and collect a sample of the acid solution. Note the flow totalizer reading on the regenerant waste line, and set the automatic sampler to begin collection from the regenerant waste line. Start the regeneration of the cation column, and reset the system to allow feed rinse wastewater to be processed using the alternate set of columns.
- 5. At the conclusion of the cation regeneration mode, set the system to "pause", note the acid solution flow totalizer reading. Stop the automatic sampler, and retrieve the accumulated sample. Open column top port and collect sample of cationic resin.
- 6. Before the anion regeneration begins, note the caustic solution flow totalizer reading and collect a sample of the basic solution. Set the automatic sampler to begin collection, and start the regeneration of the anion column. Reset the system to allow rinse wastewater to continue being processed using the alternate set of columns.
- 7. At the conclusion of the anion regeneration mode, set the system to "pause", note the caustic solution flow totalizer reading and the flow totalizer reading on the regenerant waste line. Stop the automatic sampler, and retrieve the accumulated sample. Open top port and collect sample of anionic resin.
- 8. Reset the system to allow rinse wastewater to continue being processed using the alternate set of columns.

#### C. DATA VALIDATION AND ASSESSMENT

#### 1. Data Review and Validation

Data Review and Validation will be conducted by members of the Project Team in order to examine the results of field test methods, analytical tests, and surveys to determine if data supports Hydromatix's claims, and to ensure that the procedures and activities conform to the requirements outlined in the Workplan. Dr. Ruth Chang and Mr. Ed Benelli will verify the procedures and data generated by Advanced Technology Laboratories and Purolite Laboratory. Dr. Bruce La Belle, Mr. Ed Benelli and others from the Project Team will provide qualitative review of survey results to ensure that data can support the project evaluation.

Data generated through surveys will be validated by the Project Manager in a number of ways:

- telephone calls to end-users;
- on-site facility observations;
- review of facility waste generation and management records or manifests; and
- review of facility's waste analysis documentation

If Hydromatix obtains new end-users during Workplan implementation and field testing, they

will be contacted to participate. For those willing, the questionnaire will be sent to them to complete and return.

No deviations from the sampling design are anticipated. If any occur, they will be documented and the effects on the data usability will be evaluated. If any of the following conditions occur, then the sample laboratory analysis will be considered void and the resulting data disregarded:

- known sample contamination occurred;
- QA/QC procedures were not followed; or
- results of percent recoveries or charge balance calculations are recognized to be unacceptable.

Sample preservation, analytical methods, and data integrity will be validated by the laboratory chemist, analyst, and supervisor, and will be documented in the laboratory data packages.

# 2. Quality Control Requirements

For samples sent to ATL, QA/QC will be performed in accordance with internal laboratory procedures. For each set of water samples sent to ATL, a blank and a field spike will be included for analyses. The travel blanks will consist of deionized water. The field spike will be prepared at DTSC's Southern California Hazardous Materials Laboratory, and will consist of a known synthetic mixture of cations and anions expected to be present in the waste samples. ATL will also analyze laboratory spikes, duplicates, and blanks per their QA procedures. QA/QC for the samples sent to Purolite's analytical laboratory will be performed in accordance with their internal company procedures. Quality control cation and anion resin samples of fresh unused materials will be submitted for resin analyses along with the actual resin samples collected during the sampling events. No spike will be prepared for the resin samples. Spike samples of known concentrations of both acid and base solutions will be submitted to ATL along with actual samples collected during field sampling events.

Accuracy, precision and detection limit ranges listed by ATL for the analyses to be done are:

| metals | 80-120% | 20% | 0.5-1 μg/L (Cu, Ni, Zn) ATL Instrument DL   |
|--------|---------|-----|---|
| ions   | 85-115% | 15% | PO <sub>4</sub> <sup>-2</sup> .2 mg/L<br>F-,Cl <sup>-</sup> .5 mg/L<br>SO <sub>4</sub> <sup>-2</sup> , NO <sub>3</sub> <sup>-2</sup> 1.5 mg/L |

For the spiked samples analyzed by ATL, an acceptable percent recovery range will be defined as 80 to 120 percent. For the duplicate samples analyzed by ATL, an acceptable precision range will be defined up to 20 percent. Samples analyzed with associated spike recoveries outside of this range will not be used in subsequent calculations of mass balance, acid or base consumption,

or resin capacity restored. Samples which fail charge balance criteria will similarly be disregarded. Fresh resin samples will be similarly used to monitor the analysis performed at Purolite Laboratory. Recoveries not within the range of 80 to 120 percent shall be deemed unacceptable and will render the associated data unusable.

Calibration and re-calibration procedures conducted on the flow sensors will be used to verify an acceptable level of accuracy for the units. The units are measured at the manufacturer's facility at ten flowrates. The acceptable range for both the new and used units shall be defined as no flowrate measurement exceeding one percent deviation.

# 3. Documentation and Record Keeping

DTSC will follow established procedures for maintaining the control and integrity of samples from collection, preservation, and transportation. Resin samples will be shipped under chain of custody to Purolite Co. directly for analysis. For the various water samples, Advanced Technology Laboratories also has established procedures for maintaining the control and integrity of samples from collection, preservation, transportation, and throughout the laboratory services, storage, and disposal.

All documentation and records produced during sample collection, packaging, transportation, chain-of-custody, lab analysis and sample control, to final reporting, will be filed with the appropriate laboratories and copies will be kept by the Project Manager.

All questionnaire, telephone, and onsite observation records will be kept by the Project Manager in hard copy format. Reports generated by the Project Manager will be kept by the Project Manager in hard and electronic formats. Electronic reports will be copied to diskette for back-up and kept by the Department's Assignment Manager.

All reports and data generated by this project will be centrally filed with OPPTD for a minimum of three years following verification. At this time, the Department's Assignment Manager will have the authority to transport the report and supporting data to State Archives.

# 4. Reconciliation with Data Quality Objectives

Data from regenerant waste flowmeters and resin testing will be used to determine the main data quality objectives of specific volume and resin capacity restored. The specific volume will be correlated with the resin capacity restored. Using the capacity data, the regenerant waste volumes measured for the anion and cation columns will be normalized to 100 percent restoration. Resin analysis will also yield the current operating capacity and provide a typical resin lifespan for similar applications. The range, average, and confidence interval for the specific volume and resin capacity restored will be determined from the four runs.

The capacities restored to the columns will also be checked by performing a mass-balance calculation. The ions in the incoming rinse wastewater, product DI water, and regenerant waste will be balanced. These calculations will be used to support the capacity restored results

obtained through resin analysis. Charge balances will be done on samples where complete analyses were performed. In the possible case of incomplete mass or charge balances, that data will not be used in subsequent calculations, and an explanatory note will be added to the text of the final report.

The rinse wastewater volume treated will be measured by the flow totalizer; the range, average, and confidence interval will be calculated and presented.

Analytical concentrations of the acid and base solutions will also be measured. The range, average, and confidence interval of the four samples will be calculated and tabulated.

Factors which may affect performance include:

- Age of the resins
- Untreated rinse wastewater characteristics
- Untreated rinse wastewater flow rate

Both Hydromatix, and the host facility Aero-Electric Connector, specify a desired flow rate for the system of approximately 35 gpm, which is currently monitored and controlled with a simple rotameter and ball-valve arrangement. A higher degree of system performance is achieved with a constant flow rate. With the specified the equipment, the current flow rate can be easily displayed by switching modes on the totalizers. The acceptable range for both the new and used flow sensors shall be defined as no flowrate measurement exceeding one percent deviation. Exceeding this range may result in a rejection of that data.

Field and laboratory measurements will be used to determine if Hydromatix's specific volume of regenerant waste claim is met at the 90% confidence level. The probability of making a decision error is low, due to the high accuracy and precision of the analytical methods to be used and the number of samples collections planned. The negative effects of making either a false positive or false negative decision error will be low, due to (1) the ion exchange system will continue to perform its function successfully, but a higher or lower regeneration frequency, and (2) there will be no regulatory, health, or safety effects from a decision error. In the case of higher or lower regeneration frequency, the cost an end-user incurred for regeneration chemicals would be more or less than they expected.

The data and information resulting from field and laboratory testing will be reviewed by the Project Manager and the Project Review Team. If the collected data and supporting information are sufficient to verify the technology, the Department will prepare a proposed Verification Decision, our recommendation to verify the technology, and a Technology Evaluation Report for U.S. EPA review. If the provided data and supporting information are insufficient to verify the technology, the Department will discuss other options with Hydromatix such as a narrower scope of verification or additional field tests. If Hydromatix wants to alter the scope of their performance claims the Department will provide advice regarding their alteration, such as the likelihood of successful verification and/or if additional field test(s) will be necessary, and determine if such a scope is representative of a potential commercial application. If the collected data and supporting information are sufficient to verify the technology under a narrowed scope,

| the Department will prepare an amended Verification Decision proposal and Technology Evaluation Report for U.S. EPA review. |  |  |
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# **APPENDIX A - Hydromatix System Description**

To understand the difference between the 786E series systems and conventional systems, it is important to understand the steps required achieving a good "regeneration" of the resins. In a two-bed deionizer, there is a cation column, which needs to be regenerated with a strong acid like hydrochloric or sulfuric, and there is an anion column, which needs to be regenerated by a strong alkali like sodium hydroxide. Since the procedure is the same for both columns, except for the chemicals used, the following explanation will only cover the cation regeneration. Each step of the regeneration is explained including the volume of water required (this data is substantiated by a typical resin data sheet provide by a resin manufacturer) and how the 786E series recycling steps are accomplished.

**BACKWASH:** This is usually the first step in the conventional ion-exchange systems. In this cycle, the resins bed is lifted to flush out the dirt and debris collected by the resins during the service cycle. The conventional ion exchange column has 40% freeboard volume above the resin beds to allow the resin to expand. For this system, this cycle will generate 10-25 gallons of waste per cubic foot of resin. The 786E systems use a packed bed (with no freeboard above the resin beds) by which this wastewater volume is eliminated. Unlike the conventional systems, the 786E systems utilizes very good filtration upstream of the ion exchange columns such that backwash is not required. However, because resins are round beads, even a packed bed has void volume filled with process water, which occupies the bed during the previous service cycle. This void volume is 40% of the resin volume. Hence, both packed bed and conventional systems would store 0.4 cubic foot of clean water per cubic foot of the resin beds. This void volume is discharged to waste by a conventional ion exchange system. However, the 786E systems are designed such that during the next cycle (chemical draw), this void clean water from the column is saved and is diverted back to the feed tank via a PLC controlled value.

<u>CHEMICAL</u>: After backwash, the resins are subjected to several gallons of acid (for cations) or caustic (for anions). The chemicals allow the resins to exchange the ions removed during the service cycle for hydrogen (cation) or hydroxide (anion) ions. The waste generated from this cycle is the only waste 786E Systems generate and is typically 10-12 gallons per cubic foot of resin. This waste is sent to the neutralization tank.

**SLOW RINSE:** Once a sufficient amount of chemical has been fed to the resins, clean water is fed at a slow rate to rinse the excess acid and caustic out from the columns. This cycle is typically run at 0.5 gpm per cubic foot for 60 minutes (or 30 gallons of waste per cubic foot of resin bed). Conventional systems discard this waste. Since this rinse contains a dilute form of the regenerating chemical, the 786E Systems save the solution from this entire cycle by using proprietary distribution design and resin bead size. The 786E systems rinse out from this cycle is 10-12 gallons per cubic foot. These solutions are sent to the acid and base makeup tanks for readjustment of their concentrations and subsequent reuse during the next regeneration cycle.

**<u>FAST RINSE:</u>** The slow rinse is followed by a fast rinse to remove any hidden pockets of chemical that might remain. This step generates 30 gallons per cubic foot of waste in

conventional systems. However, the 786E Systems run the fast rinse through the exhausted anion column and fill the rinse tank, preserving all of the water this rinse would generate in a conventional system.

**FINAL RINSE:** After the above cycles are repeated for the cation and the anion, the entire system is rinsed to eliminate all remaining chemical until a desired conductivity is achieved. Typically, this water is discharged to drain by conventional systems. The 786E Systems recirculate this rinse through the system, such that the sodium in the effluent is removed by the cation bed and exchanged for H<sup>+</sup> turning the NaOH to H<sub>2</sub>O. Once again a significant quantity of water, which ends up in the drain with conventional technology, is reused and recycled.

Using a combination of the above and other patented technologies, the 786E Systems are able to cut down the regenerant waste volume by 95% or more, and intrinsically offer the following additional benefits:

- The 786E Systems consume 25%-40% less acid and caustic during regeneration, compared to conventional systems. This results in operating costs that are lower than complicated combination technologies like electrowinning, R/O, or Metal Selective Resin Columns.
- Since the regenerant waste has to be treated before discharge or evaporation, the cost of such treatment will be significantly lower with the 786E System.
- Due to the reduced volume, the regenerant waste from the 786E System can be batch-treated to ensure compliance. The high volume of waste from a conventional system would have to be treated by a continuous duty precipitation system, with the possibility of violations due to upsets.
- The 786E Systems are regenerated with premixed acid and caustic. No eductors are used during regeneration. The result is precise and predictable regenerations.

# **APPENDIX B - Hydromatrix End-User Questionnaire Form**